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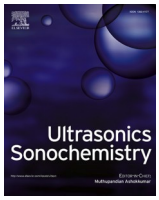

Sonochemical degradation of CCl_4 in alcohol-water system☆

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ABSTRACT

In this study, methanol (MeOH), ethanol (EtOH), 1-propanol (PrOH), and ethylene glycol (EG) were used as alcohol-CCl_4 pretreatment solutions for the first time to investigate the effect of alcohol on the sonochemical degradation of CCl_4. The results showed that this pretreatment method provided the homogeneous dissolution of CCl_4 in aqueous solution in a short preparation time and the enhancement of the sonochemical degradation of CCl_4. In addition, the rate of the degradation of CCl_4 related to the hydrophobicity of the added alcohol. It was considered that high hydrophobic alcohol molecules accumulated at the interface region of cavitation bubbles contributed to the scavenging of •OH radicals produced by sonolysis of water and to the pyrolysis itself, resulting in the production of a large amount of •H radicals. The formation of •H radical and its secondary products, such as H_2 could lead to the reductive degradation of CCl_4. In previous studies, the addition of alcohol has been observed only as a negative effect on the sonochemical degradation of organic compounds such as the reduction of bubble temperature, and the quenching of the oxidative degradation by scavenging •OH radicals. However, our results indicated that the positive effects of alcohol on the sonochemical degradation of CCl_4 were more significant than the negative effects.

1. Introduction

Ultrasonic technology has been widely used in various fields, such as food science, biomedical, physical, and chemical applications [1,2]. The ultrasonic irradiation of an aqueous solution can lead to the formation and growth of cavitation bubbles due to the alternating compression and rarefaction induced by acoustic waves. The accumulated energy releases upon the collapse of these cavitation bubbles as transient high temperatures and high pressures [3]. In addition, highly reactive radicals, such as •H radicals, and •OH radicals were formed by the pyrolysis of water molecules during cavitation [4–6]. This is the core phenomenon of ultrasonic water treatment technology.

As a new environmental technology, aqueous solutions of organic compounds, such as azo dyes [7], chlorophenols [8], benzene, and other aromatic chlorinated compounds [9] have been effectively degraded by ultrasonic irradiation. The mechanism of sonochemical degradation is also discussed. For example, the oxidation caused by •OH radicals is one of the main pathways that organic compounds eliminated in water [10,11]. In addition, volatile organic compounds (VOCs) tend to degrade via pyrolysis. This is because VOCs volatilize easier and directly pyrolyze at the inside of cavitation bubbles where reaches more than thousands of degrees. On the other hand, VOCs and their gaseous byproducts diffuse into the interior of cavitation bubbles leads to the change in the gas composition inside the bubbles [12–16]. The specific heat ratio (γ) of a certain gas inside the bubbles directly affects the maximum temperature achieved by cavitation bubbles collapse, as shown in eq. (1). In this equation, T_{max} is the maximum temperature of bubble upon collapse, T_0 is the temperature of the bulk solution, P_0 is the pressure in the bubble at its maximum size, P_{max} is the maximum pressure of the bubble upon collapse, and γ is the specific heats ratio (C_p/C_v) [17,18].

$$T_{max} = \frac{T_0 P_{max}(\gamma - 1)}{P_0} \quad (1)$$

Carbon tetrachloride (CCl_4), a typical harmful VOC, is widely present in the environment also as a greenhouse gas [19]. The simple chemical structure of CCl_4 makes it suitable for the analysis of physicochemical characteristics of cavitation bubbles and the reactions of sonochemical degradation. In decades, the sonochemical degradation of CCl_4 in aqueous solution has been investigated under different frequencies, saturating gases, initial concentrations of CCl_4 and temperatures of bulk solution [20–24]. Other enhancement methods have also been explored

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by Hung *et al.* [25]. They demonstrated that the rate of ultrasonic degradation of CCl_4 can be enhanced in the combination system of ultrasound and Fe^0, where the FeO surface of Fe powder is removed by shockwave from cavitation bubbles and thus the reductive degradation of CCl_4 proceeds in addition to conventional pyrolysis reactions. In this paper, we first used the alcohol-CCl_4 pretreatment method. In addition to the rapid and efficient preparation of a homogeneous aqueous solution of CCl_4, we successfully accelerated the rate of the sonochemical degradation of CCl_4. The effect of alcohol on the sonochemical degradation of CCl_4 was discussed.

2. Experimental

All water used in this study was treated by a Millipore Direct-Q 3UV system. The reagents involved in this study were listed as follows: carbon tetrachloride, CCl_4, 99.8 % (Wako); methanol, MeOH, 99.9 % (Sigma-Aldrich); ethanol, EtOH, 99.5 % (Fujifilm Wako); 1-propanol, PrOH, 99.5 % (Fujifilm Wako); ethylene glycol (EG), 99.5 % (Wako); Na_2CO_3 (Wako); $NaHCO_3$ (Wako); H_2SO_4 (95 %, Fujifilm Wako); argon (Ar), ZERO-A 99.999 %, (Sumitomo Seika Chemicals). All of the experiments were conducted after saturating the solutions with Ar for 20 min to obtain the highest theoretical bubble temperature.

CCl_4 is barely soluble in water but miscible in organic solvents. The solubility of CCl_4 in water is around 5.2 mM at 20 ℃ [26,27]. Based on this property, four types of CCl_4 aqueous solution were prepared and irradiated by ultrasound in this study. The experiments included (E1) the irradiation immediately after placing CCl_4 in Ar-saturated water, where CCl_4 was incompletely dissolved; (E2) the irradiation after dissolving CCl_4 completely in Ar-saturated water; (E3) the irradiation after adding a certain amount of alcohol to the solution where CCl_4 was completely dissolved in Ar-saturated water as (E2); (E4) the irradiation of alcohol-CCl_4 pretreatment solution in Ar-saturated water. The initial concentration of CCl_4 in the solutions mentioned above was designed to be the same. Details of the reagent preparation are presented in Supplementary Data. Methanol, ethanol, 1-propanol and ethylene glycol were used as solvents in the pretreatment solutions in this study. The error caused by the difference of initial concentration of CCl_4 (1035 ± 5 μM) was negligibly small.

In a water bath that was maintained at 20 ± 1 ℃ by a cold-water circulation system (TAITEC CP-150R), the aqueous solution of CCl_4 prepared by the above methods was placed in a cylindrical glass reaction vessel. The vessel was placed at 4.0 mm height from the surface of ultrasonic transducer to reduce ultrasonic wave reflection. The solution (100 ± 0.5 mL) was irradiated at the frequency of 200 kHz by an ultrasonic generator (Kaijo, type 4021, Lot. No. 11EA00103) and an ultrasonic transducer (Kaijo, type 4611, Lot. No. 00102) at power dial of 8. The actual calorimetric power was measured to be 13.6 W. The details of the calorimetry are presented in Supplementary Data. In our sonication system, it was confirmed that the temperature of collapsing bubbles was estimated to reach around 3500 K, based on the analysis of the kinetics of methyl radical recombination reactions in the sonolysis of aqueous t-butanol solution under Ar [4]. Around 1.0 mL of the liquid sample was collected into a 13.5 mL screw tube bottle (AS-ONE) after required irradiation time. Since Cl^- is formed from the degradation of CCl_4, the amount of Cl^- divided by 4 referred to the degradation amount of CCl_4. The concentration of Cl^- in the sample was analyzed by ion chromatography (761 compact IC, Metrohm). The IC analysis conditions were as follows: the separation column was Shodex IC SI-90 4E, the eluent was a mixture of aqueous solution of 1.8 mM of Na_2CO_3, 1.7 mM of $NaHCO_3$, and 5 vol% of methanol at a flow rate of 1.00 mL/min. The addition of methanol to the aqueous eluent accelerates lipophilic property, allowing the IC system to be rinsed by the eluent even during samples testing. The regeneration solution was 40.0 mM of H_2SO_4 in aqueous solution. The gaseous samples were collected by a 1 mL microsyringe (ITO MS-GAN100) after required irradiation time. The yield (μmol) of gaseous product was divided by solution volume to express in μM. This calculation makes it convenient to compare with the results of the concentration of sample solution. The yield of H_2 was analyzed by a gas chromatograph (Shimadzu, GC-2014AT, TCD model). The GC-TCD analysis conditions were as follows: the separation column was Molecular Sieve 5A, the column temperature was 50 ℃, the detector temperature was 150 ℃, the carrier gas was Ar at a flow rate of 40 mL/min, the reference gas was Ar at a flow rate of 40 mL/min, and the sample injection amount was 0.50 mL.

The measurement of sonoluminescence (SL) intensity was conducted under the same experimental conditions as above: In a 20 ℃ water bath, Ar-saturated aqueous solutions of alcohols (100 ± 0.5 mL) were irradiated at 200 kHz with the same equipment, while the SL intensity was measured. The cylindrical glass reaction vessel and water bath wall both have quartz window parts. The SL spectrum generated during sonication was measured by a fluorescence spectrophotometer (Shimadzu, RF-6000), where the slit width was set at 20 nm. The scan speed was 200 nm/min. Before SL measurements, a background spectrum without ultrasonic irradiation was acquired and this value was subtracted from the SL spectrum.

3. Results and discussion

3.1. Sonochemical degradation of CCl_4 and the effect of homogeneity

The effect of EG addition on the sonochemical degradation of CCl_4 in aqueous solution was investigated. Four types of aqueous solution of CCl_4 as described in experimental section as (E1), (E2), (E3), and (E4) were prepared and irradiated by ultrasound. All the experiments were repeated three times. Fig. 1 shows the degradation of CCl_4 in aqueous solution as a function of the sonication time. In Fig. 1a, within 5 min of sonication, in the absence of EG, only 14.65 % of CCl_4 was degraded where CCl_4 was added just before irradiation as (E1). It was increased to 61.89 % while 60 mM of EG was used as solvent in pretreatment solution (EG-CCl_4) as (E4). It is believed that this remarkable enhancement is mainly attribute to the homogeneity of CCl_4 aqueous solution through EG-CCl_4 pretreatment method. Although the solubility of CCl_4 in aqueous solution is reported to be 5.2 mM, when 1.0 mM of CCl_4 was used, a large amount of CCl_4 remained as sediment at the bottom of the reaction vessel before irradiation owing to its low dissolution rate. The low bulk concentration of CCl_4 in such a heterogeneous solution should lead to the low degradation rate. However, the contribution of EG is not limited to the effect of homogeneity. In Fig. 1b, CCl_4 was completely dissolved in water to prepare homogeneous solution before sonication. The sonication of the homogeneous aqueous solution of CCl_4 in the absence of EG as (E2) and presence of EG as (E3) is presented. After 5 min of ultrasonic irradiation, the degradation of CCl_4 was enhanced from 58.93 % to 63.42 % with the addition of 60 mM of EG. The 4.49 % of the enhancement is considered as the result of the positive effect of EG addition.

3.2. Effect of alcohol on the degradation of CCl_4

The effect of aliphatic alcohol, such as MeOH, EtOH, PrOH and diol, such as EG on the sonochemical degradation of CCl_4 were individually investigated. In order to exclude the effect of homogeneity, all the experiments were conducted as (E4) and repeated three times. Fig. 2 shows the comparison of degradation rate of CCl_4 within 5 min of ultrasonic irradiation between EG-CCl_4, MeOH-CCl_4, EtOH-CCl_4, and PrOH-CCl_4 pretreatment solution in aqueous solutions as a function of the concentration of alcohol. Among them, 30 mM was the minimum available concentration of a homogeneous EG-CCl_4 pretreatment solution. Because of the strong polarity of diol, CCl_4 could not dissolve in low volume of EG. At all alcohol concentrations from 0 mM to 60 mM, the sonochemical degradation of CCl_4 was accelerated with the increasing concentration of alcohol. In addition, the rate of degradation of CCl_4 was in the order of EG-CCl_4 < MeOH-CCl_4 < EtOH-CCl_4 < PrOH-CCl_4, which

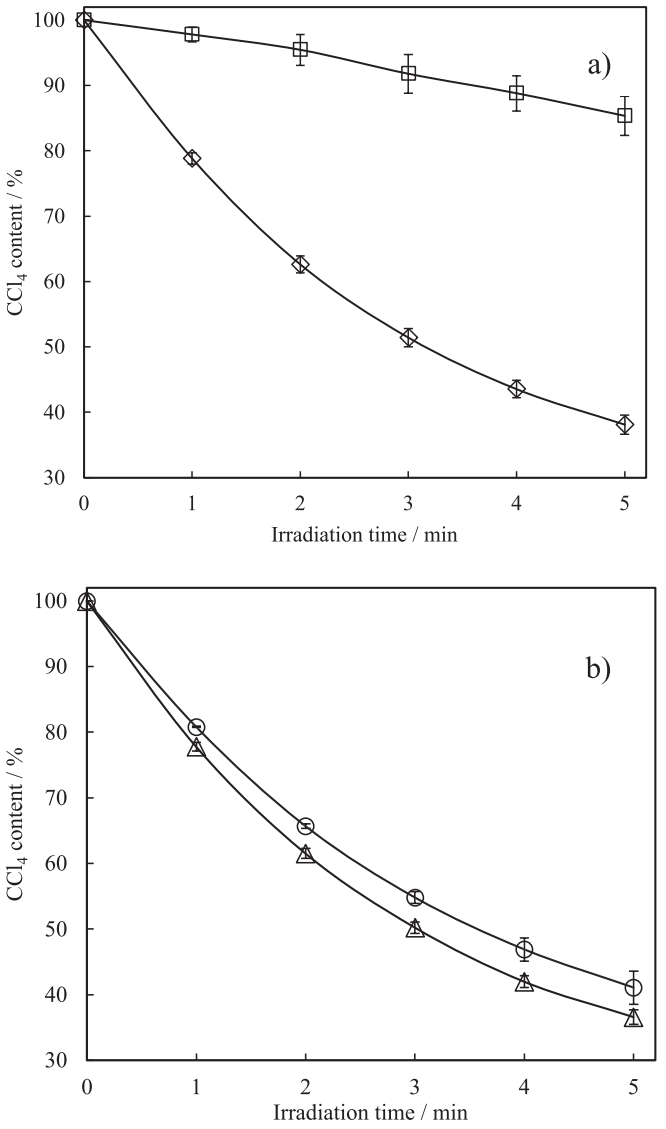

Fig. 1. (a) Degradation of (E1) CCl_4 (incomplete dissolution) in aqueous solution (□), and (E4) 60 mM EG-CCl_4 in aqueous solution (◇). (b) Degradation of homogenous aqueous solution of CCl_4 (E2) in the absence (○), and (E3) presence (△) of 60 mM EG.

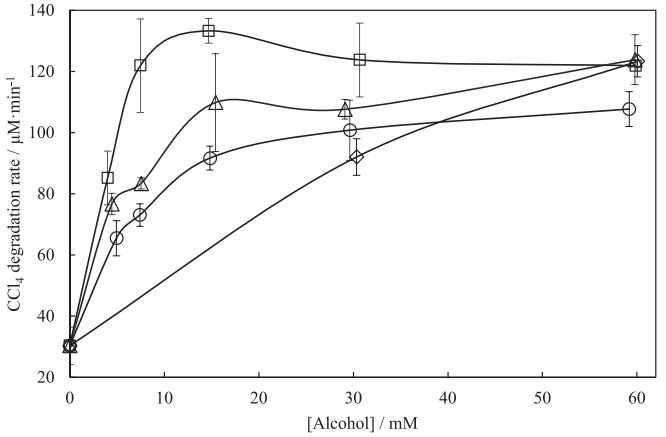

Fig. 2. Rate of CCl_4 degradation in different concentration of PrOH-CCl_4 (□), EtOH-CCl_4 (△), MeOH-CCl_4 (○), and EG-CCl_4 (◇) in aqueous solution.

was the same order with their hydrophobicity. Log *P* represents the octanol–water partition coefficient and is commonly used as a measure of the lipophilicity or hydrophobicity of a certain compound. The log *P* value of alcohol used in this study was as follows: EG (log $P = -1.36$) < MeOH (−0.77) < EtOH (−0.31) < PrOH (0.25) [28]. It is believed that alcohol molecules with higher hydrophobicity are likely to accumulate at the interface region of cavitation bubbles, where the concentration of •OH radicals and the temperature are higher than those in the bulk solution. As shown in Eqs. (2)–(5), alcohol both scavenges •OH radicals produced by sonolysis of water [29] and undergoes pyrolysis itself [30], resulting in the production of a large amount of •H radicals. The formation of •H radicals and its secondary products, such as H_2 could lead to the reductive degradation of CCl_4.

$$H_2O \xrightarrow{)))} \bullet H + \bullet OH \quad (2)$$

$$RHOH + \bullet OH \rightarrow \bullet ROH + H_2O \quad (3)$$

$$RHOH \xrightarrow{)))} \bullet H + \bullet ROH \quad (4)$$

$$2 \bullet H \rightarrow H_2 \quad (5)$$

Fig. 3 shows the relative SL intensity of aqueous solutions of EG, MeOH, EtOH, and PrOH from 290 to 600 nm, where the SL intensity of pure water under the same experimental condition was defined as the reference value of 1. As shown in Fig. 3, relative to water, quenching of SL occurred in 30 mM aqueous solutions of all alcohols, where the SL intensity was in the order of PrOH < EtOH < MeOH < EG. This is the same order as the hydrophobicity of alcohols discussed above. The consumption of •OH radicals by hydrophobic alcohol molecules accumulated at the interface region of cavitation bubbles leads to a weakening of the spectral peak intensity from •OH radical emission at around 310 nm [31], as shown in Fig. S1 in Supplementary Data. In addition, in the same alcohol-water system, the order of SL intensity and the degradation rate of CCl_4 presented in Fig. 2 showed opposite trends. Therefore, the rate of the sonochemical degradation of CCl_4 in alcohol-water solution could not be enhanced by the change in physical cavitation behavior induced by the presence of alcohol.

Fig. 4 shows the yield of H_2 via water sonolysis in the presence of alcohol (Fig. 4a) and alcohol-CCl_4 (Fig. 4b). As shown in Fig. 4a, while 30 mM of alcohol was added in water, the yield of H_2 after 10 min of ultrasonic irradiation was in the order of addition alcohol of EG < MeOH < EtOH < PrOH. This order verified that sonolysis of aqueous solution of high hydrophobic alcohol produces more amounts of •H radical or H_2 as

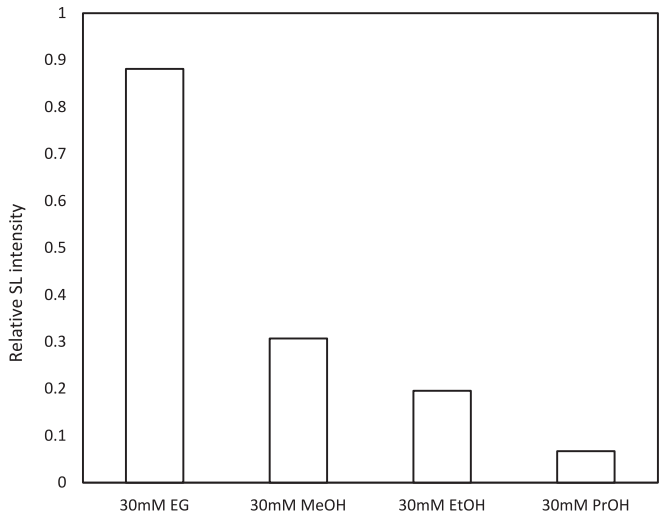

Fig. 3. SL intensity of 30 mM EG, 30 mM MeOH, 30 mM EtOH, and 30 mM PrOH in aqueous solutions, relative to pure water sonication.

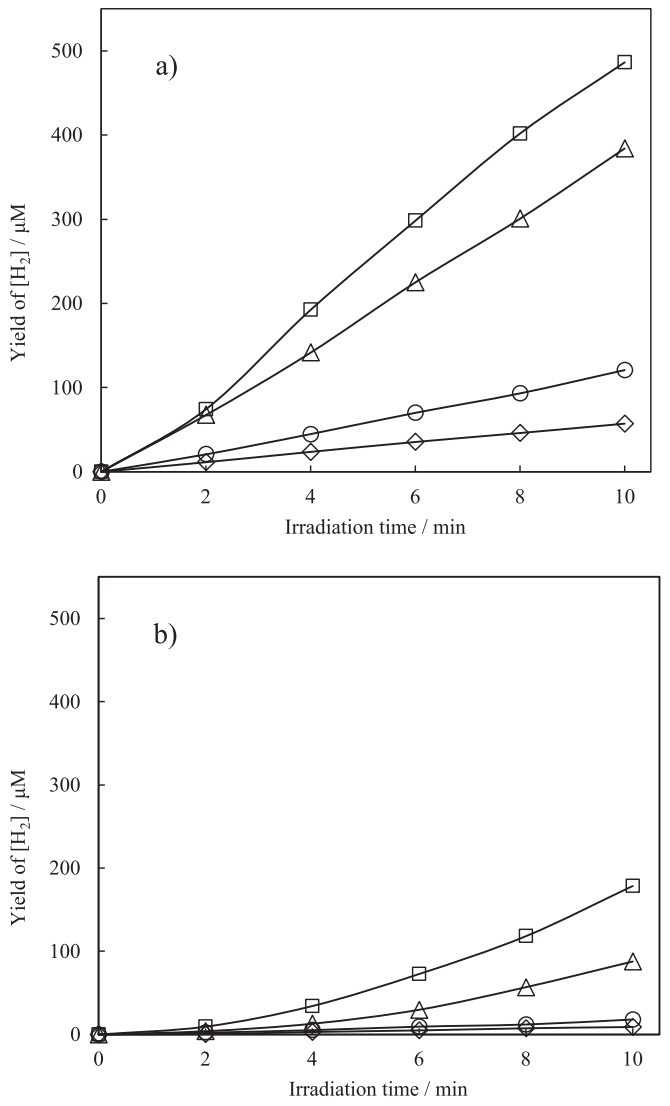

Fig. 4. (a) Yield of H_2 from sonolysis of aqueous solution of 30 mM PrOH (□), 30 mM EtOH (△), 30 mM MeOH (○), and 30 mM EG (◇). (b) Yield of H_2 from sonolysis of aqueous solution of 30 mM PrOH-CCl_4 (□), 30 mM EtOH-CCl_4 (△), 30 mM MeOH-CCl_4 (○), and 30 mM EG-CCl_4 (◇).

discussed above. Fig. 4b indicates that the yield of H_2 decreased by more than 60 % in the presence of CCl_4. Moreover, the decreasing amount of H_2 correlated to the rate of the sonochemical degradation of CCl_4. At 30 mM of alcohol, the reduction in the amount of H_2 followed the same order as the degradation rate of CCl_4 presented as in Fig. 2, where EG-CCl_4 < MeOH-CCl_4 < EtOH-CCl_4 < PrOH-CCl_4. This result further suggested that •H radical or H_2 generated by sonolysis of water-alcohol system reacts with CCl_4 under ultrasonic irradiation. An increase in the concentration of •H radical or H_2 would result in a faster sonochemical degradation rate of CCl_4.

So far, it has been reported that most of the organic compounds are sonochemically degraded via oxidation caused by •OH radicals. Therefore, the •OH radical scavenging effect of alcohol could lead to the quenching of the oxidative degradation of organic compounds. For example, in the results of Nagata *et al.* on the sonolysis of chlorophenols, around 60 % of the degradation of 3-chlorophenol (3-CP) was quenched by adding 1 mM of t-BuOH under Ar [32]. Another example, as shown in Fig. S2 in Supplementary Data, is that the amount of methyl orange (MO) degraded after 10 min of ultrasonic irradiation decreased from 57.7 % to 24.8 % with the addition of 60 mM of EG. However, our results indicates that the pathway of sonochemical degradation of CCl_4 would not involve the •OH radical-induced oxidation. Instead, the trigger of the sonochemical degradation of CCl_4 is believed to consisted of two parts. One is the thermal energy produced by cavitation, which is commonly suggested by most of the researchers [33]. The other is the •H radical-induced reduction, as suggested by the addition of alcohol in this study.

On the other hand, it is possible that the addition of alcohol negatively affects the sonochemical degradation process from a physicochemical perspective of cavitation bubbles. For example, an increase in viscosity of the aqueous solution induced by alcohol addition leads to the suppression of the bubble expansion [34] and increase of the cavitation threshold [35], where the effects of high EG concentrations (10–100 mass%) were investigated. However, the effect of viscosity in this study is insignificant due to the low alcohol concentration. The relative viscosities (η/η_0) of aqueous alcohol solutions with respect to water at 20 ℃ are pure water (1.000), 1 mass% MeOH (1.038), 1 mass% EtOH (1.044), 1 mass% PrOH (1.049), and 1 mass% EG (1.018) [36]. In comparison, the highest concentrations used in this study, 60 mM MeOH, EtOH, PrOH, and EG, correspond to 0.192 mass%, 0.276 mass%, 0.361 mass%, and 0.372 mass%, respectively, resulting in much smaller changes in bulk viscosity than the 1 mass% reference value mentioned above.

In addition, it is also known that the presence of alcohol in water affects the surface tension of the solution. At 20 ℃, the surface tensions of aqueous alcohol solutions in literature [37,38] are in the order of pure water (72.75 mN/m) > 5 mass% EG (70.7 mN/m) > 5 mass% MeOH (63.46 mN/m) > 5 mass% EtOH (56.41 mN/m) > 5 mass% PrOH (42.51 mN/m). This is because the more hydrophobic the alcohol, the lower its surface tension. It has also been reported that the surface tension of 55 mM PrOH solution is 68.2 mN/m [39]. Based on these surface tension values, in this study, the surface tension variations of the solutions caused by 4–60 mM of alcohols is considered negligible. Therefore, the effect of the change in the surface tension on the cavitation would be very small.

Moreover, the accumulation of hydrophobic alcohol molecules at the interface region of the bubbles not only promotes the reduction of CCl_4 by •H radicals or H_2, but also negatively affects its pyrolysis by consuming the pyrolytic energy and decreasing the temperatures generated upon the collapse of cavitation bubbles. The gaseous pyrolysis products of alcohol with lower γ value, such as CO, CO_2, H_2, enter the interior of cavitation bubbles, consequently resulting in the decrease of the bubble temperature [40–42]. The γ value of the gas related to this study at 15 ℃ is as follows: Ar (1.67), H_2 (1.41), CO (1.40), CO_2 (1.30), CCl_4 (1.11) [43]. Although this argument is consistent with the quenching of sonoluminescence observed in this study, it is considered that the bubble temperatures estimated by the kinetics of methyl radical recombination reactions are not significantly affected when low concentrations of alcohols are used. In the study of Ashokkumar *et al.* [44], at ethanol concentrations from 0 mM to 100 mM in Ar-saturated aqueous solutions, the measured maximum bubble temperatures remained within 4200 ± 200 K at the irradiation frequency of 365 kHz, while the SL intensity was quenched extremely. Consequently, our results demonstrated that the positive effects of alcohols on the sonochemical degradation of CCl_4 are more significant than their negative effects.

4. Conclusion

In this study, we first used MeOH, EtOH, PrOH and EG as solvents in the pretreatment solutions (alcohol-CCl_4) to investigate the effect of alcohol on the sonochemical degradation of CCl_4. By using this method, CCl_4 could completely dissolve in an aqueous solution within a short preparation time. The homogeneity of CCl_4 in aqueous solution provided by an alcohol-CCl_4 pretreatment solution was considered as one of the main reasons of the enhancement of the degradation of CCl_4. In addition, the rate of degradation of CCl_4 was in the order of EG-CCl_4 < MeOH-

CCl_4 < EtOH-CCl_4 < PrOH-CCl_4, which was the same order with their hydrophobicity. It was considered that alcohol molecules with higher hydrophobicity accumulated at the interface region of cavitation bubbles, scavenged •OH radicals produced by sonolysis of water and underwent pyrolysis itself, resulting in the production of a large amount of •H radicals. The formation of •H radicals and its secondary products, such as H_2 could lead to the reductive degradation of CCl_4. This was also suggested by analyzing the yield of H_2 from sonolysis of alcohol and alcohol-CCl_4 pretreatment solution in aqueous solution. In the presence of CCl_4, the yield of H_2 decreased and the reduced amount of H_2 correlated to the rate of the sonochemical degradation of CCl_4.

So far, it has been reported that the addition of alcohol mainly has negative effects on the sonochemical degradation of organic compounds, such as decreasing the bubble temperature, and quenching the oxidative degradation as an •OH radical scavenger. However, our results indicated that the addition of alcohol accelerated the rate of the sonochemical degradation of CCl_4. The positive effects of alcohol were more significant than the negative effects.

CRediT authorship contribution statement

Aerfate Abulikemu: Writing – review & editing, Writing – original draft, Investigation. **Kenji Okitsu:** Writing – review & editing, Writing – original draft, Supervision, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ultsonch.2025.107581.

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